STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF

THE FLASH POINT OF LIQUIDS BY SETAFLASH (SMALL SCALE) CLOSED-CUP APPARATUS

(Based on SW-846 METHOD 1020B)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5 CHICAGO REGIONAL LABORATORY
536 SOUTH CLARK STREET (ML-10C)
CHICAGO, ILLINOIS 60605

TABLE OF CONTENTS

1. SCOPE AND APPLICATION: 3
2. SUMMARY OF METHOD: 4
3. ABBREVIATIONS & DEFINITIONS: 4
4. HEALTH, SAFETY & WASTE HANDLING: 4
5. CAUTIONS & INTERFERENCES: 5
6. EQUIPMENT & SUPPLIES: 6
7. REAGENTS & STANDARDS: 7
8. SAMPLE HANDLING & PRESERVATION: 7
9. SAMPLE PREPARATION & ANALYSIS: 8
10. QUALITY CONTROL: 12
11. DATA & RECORDS MANAGEMENT: 15
12. TROUBLESHOOTING: 17
13. PREVENTATIVE MAINTENANCE: 17
14. REFERENCES: 17
15. REVISION HISTORY: 18

APPENDIX A: LIMS Entry and Reporting 20

APPENDIX B: Testing High Viscosity Liquids 22

APPENDIX C: Testing Criminal Investigation Samples (CIS) 23

APPENDIX D: Deviations from the Reference Method 24

1. SCOPE AND APPLICATION:

1.1. The reference method cited is SW846 Method 1020B Revision 2 November 2004, which calls for the use of ASTM D3278-78. All method references and procedural steps in this Standard Operating Procedure (SOP) are taken from ASTM D3278-78. See Appendix D for deviations in this SOP from the reference methods cited.

1.2. This method covers the determination of the flash point, by Setaflash Closed tester, of paints, enamels, lacquers, varnishes, and related products and their components having flash points between 32 °F and 230 °F (0 °C to 110 °C) and a viscosity lower than 150 st at 77 °F (25 °C). Tests at higher or lower temperatures are possible.

1.3. This method is used to determine the characteristics of ignitability under 40 CFR part 261.21. Samples with flash points greater than 60 °C (140 °F) will only be determined as requested.

1.4. This method may also be used to determine whether a material will or will not flash at a specified temperature (i.e. flash/no flash) or to determine the finite temperature at which a material will flash.

1.5. This method should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

1.6. This method does not lend itself to method detection limit (MDL), or method reporting limit (MRL) determinations.

1.7. When using the flash/no flash method, report whether the sample flashed at the specified temperature as “flash” or “no flash”.

1.8. If a finite flash point was determined, report the mean of duplicate runs to the nearest 1 °F (0.5 °C), provided the difference between the two values does not exceed 2 °F (1 °C).

1.9. The estimate of minimum laboratory contribution to measurement uncertainty of this method is determined from the flash point of p-xylene reference material (Section 7.1). The mean of duplicate flash point determinations should be within 81 ± 1.5 °F (27.2 ± 0.8 °C). This limit is set by the reference method.

2. SUMMARY OF METHOD:

2.1. By means of a syringe, 2 mL of sample is introduced through a leak proof entry port into the tightly closed Setaflash Tester or directly into the cup that has been brought to within 5 °F (3 °C) below the expected flash point. As a flash/no flash test, the expected flash point temperature may be a specification or other operating requirement. The temperature of the apparatus is raised to the precise temperature of the expected flash point by slight adjustment of the temperature control knob. After 1 min, a test flame is applied inside the cup and note is taken as to whether the test specimen flashes or not. If a repeat test is necessary, a fresh specimen must be used.

2.2. For a finite flash measurement, the temperature is sequentially increased through the anticipated range, the test flame being applied at 9 °F (5 °C) intervals until a flash is observed. A repeat determination is then made using a fresh specimen, starting the test at the temperature of the last interval before the flash point of the material and making tests at increasing 1 °F (0.5 °C) intervals.

3. ABBREVIATIONS & DEFINITIONS:

3.1. INSTRUMENT PERFORMANCE CHECK STANDARD (IPCS) – A solvent of known flash point, such as p-xylene, used to verify that the instrument can perform to required specifications.

3.2. LABORATORY DUPLICATES (DUP) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of a number of duplicates indicate precision associated with the laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.3. FIELD DUPLICATE (FD1 and FD2) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of a number of field duplicates indicate the precision associated with the sample collection, preservation and storage, but not with the laboratory procedures.

4. HEALTH, SAFETY & WASTE HANDLING:

4.1. Users of this method should operate a formal safety program. Perform this method according to the CRL Chemical Hygiene Plan located on the CRL share drive (G:\drive).

4.2. Review SDSs for specific physical and health and hazards including appropriate PPE to be used. SDSs may be accessed at: www.sigmaaldrich.com. Alternatively, other vendor websites may be used to locate pertinent information.

4.3. General good laboratory practices are required.

4.4. Report all injuries and major spills.

4.5. **WARNING:** The operator must exercise and take appropriate safety precautions during the initial application of the test flame to the sample. Samples containing low-flash material can give an abnormally strong flash when the test flame is first applied.

4.6. **WARNING:** When using the instruments at elevated temperatures, keep hands away from the cup area, except for the operating handles as temperatures can exceed 40 °C (104 °F).

4.7. **WARNING:** Be careful in handling the cooling mixture and cooling block; wear gloves and goggles. Mixtures such as dry ice and acetone can produce severe frost bite.

4.8. Dispose of organic wastes into green labeled waste containers, and solid wastes into yellow bags labeled Hazardous Materials.

4.9. All other wastes produced in performing this method must be disposed of according to the Chicago Regional Laboratory Chemical Hygiene Plan.

5. CAUTIONS & INTERFERENCES:

5.1. Ambient pressure, sample homogeneity and drafts can affect flash point determinations.

5.2. **Caution:** Erroneously high-flash points may be obtained if precautions are not taken to avoid loss of volatile material. Do not open sample containers unnecessarily and do not transfer the specimen to the cup unless the temperature is at least 20 °F (10 °C) below the expected flash point. Discard samples in leaky containers.

5.3. **Caution:** The slide mechanism should not be pulled back too far. This will cause the mechanism to come undone. See the Primary Analyst or Group Leader to repair the slide mechanism if this occurs.

5.4. The material is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash point temperature, application of the test flame may give rise to a halo; this should be ignored.

6. EQUIPMENT & SUPPLIES[1,2]:

6.1. Recertified Support Equipment:

6.1.1. Glass Syringe: 2.0 mL ± 0.1 mL capacity at 77 °F (25 °C), to provide a means of taking a uniform specimen (ERDCO Part # RT-05520, or equivalent). Certified every two years by vendor or an outside source.

6.1.1.1. Check the capacity by discharging water into a tared small beaker or disposable solo cup and weighing. Adjust plunger if necessary. A disposable syringe of equal precision may be used. Check the glass syringe or disposable syringe daily when in use before beginning analysis.

6.1.1.2. **NOTE:** Use of a disposable syringe is recommended for samples that will be difficult to clean from the glass syringe, e.g. paint samples. BD syringe, 5 mL, Reorder No. 309603 (or equivalent).

6.1.2. Thermometer, 32 to 230 °F, minimum graduation 1 °F (ERDCO Part # RT-05531). Certified annually by vendor or an outside source.

6.1.3. Barometer, capable of reading absolute, un-corrected barometric pressure – Traceable® Barometer, Fisher Scientific Cat No. 15-079-610 (or equivalent).

6.1.3.1. Follow the certification provided by the manufacturer. Then certified yearly by vendor or an outside source after the manufacturer's certification expires.

6.1.4. Analytical balance – capable of weighing to 0.0001 g. Certified yearly by vendor or an outside source.

6.1.5. Weight set covering the range of balance use. Rice Lake Weighing Systems (or Equivalent). Certified yearly by vendor or an outside source.

6.2. Analytical Instrument:

6.2.1. Setaflash (Small Scale) Closed-Cup Tester: ERDCO Rapid Tester® Model RT-1, Type RT-00001-600 (or equivalent).

6.2.1.1. LIMS ID: Setaflash 3

[1] Refer to CRL SOP GEN026 for instructions on purchasing equipment and supplies.
[2] The following brand names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

6.2.2. Thermometer, 32 to 230 °F, minimum graduation 1 °F (ERDCO Part # RT-05531). Check the thermometer to determine that the scale error does not exceed 0.5 °F (0.25 °C). The use of a magnifying lens significantly assists in making temperature observations.

6.2.1. Cooling Block: aluminum which fits snugly within the test cup for rapid cooling of the sample cup (ERDCO Part # RT-05500, or equivalent).

6.2.2. Heat Transfer Paste (ERDCO Part # RT-05013, or equivalent).

6.2.3. Rapid Tester Butane Fuel (ERDCO Part # RT-14857, or equivalent).

7. REAGENTS & STANDARDS[3,4]:

7.1. p-xylene standard reference material (SRM) for checking the Setaflash tester (Phillips 66 grade or equivalent).

7.2. Cooling Mixture of ice water or dry ice (solid CO_2) and acetone. Purity and/or reagent grade or not applicable for the dry ice and acetone in this SOP.

7.3. To prevent materials from degradation, follow the manufacturer's recommendations and/or instructions in this SOP for preparation, handling, and storage.

8. SAMPLE HANDLING & PRESERVATION:

8.1. All samples must be collected using an approved container for the specific media. Discard samples in leaky containers.

8.2. Samples should not be stored in plastic bottles since volatile materials may diffuse through the walls of the bottle.

8.3. If available, obtain at least a 25 mL sample from the bulk source and store in a nearly full, tightly closed clean glass container or in another container suitable for the type of liquid being sampled.

8.4. Upon receipt, the sample integrity is verified. If any sample is found to be compromised, the customer must be notified in order to determine the correct action.

8.5. Holding times have not been established for this method.

[3] Refer to CRL SOP GEN026 for instructions on purchasing equipment and supplies.
[4] The following brand names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

9. SAMPLE PREPARATION & ANALYSIS:

9.1. Sampling:

9.1.1. The specimen size for each test is 2 mL.

9.1.1.1. Check the capacity of the syringe before use. See Section 6.1.1.

9.1.2. **Caution:** Erroneously high-flash points may be obtained if precautions are not taken to avoid loss of volatile material. Do not open sample containers unnecessarily and do not transfer the specimen to the cup unless the temperature is at least 20 °F (10 °C) below the expected flash point. Discard samples in leaky containers.

9.2. Preparation of Apparatus:

9.2.1. Prior to initial use or after removal of the thermometer from the Setaflash tester, insert the thermometer into its pocket with a good heat transfer paste.

9.2.2. Check the accuracy of the Setaflash tester by determining the flash point of the p-xylene SRM (Section 7.1) in duplicate (Refer to Section 10.4.1).

9.2.2.1. If not, remove the thermometer and observe whether sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup to the thermometer.

9.3. Flash/No Flash Procedure - Ambient Temperature to 230 °F (110 °C):

9.3.1. Inspect the inside of the test cup, lid, and shutter mechanism for cleanliness and freedom from contamination. Use an absorbent tissue to wipe clean, if necessary. Lock the cover lid tightly in place.

9.3.2. Switch the instrument to ON. Adjust the temperature control knob while depressing the Preset switch until the thermometer reads a temperature that is at least 5 °F (3 °C) below the specification or target flash point temperature.

9.3.3. When the digital display reaches the Preset temperature, the red light will extinguish. It may be necessary to make a slight adjustment using the temperature control knob. The red light will glow whenever the instrument is heating the cup to maintain the specified temperature.

9.3.4. Determine the current barometric pressure to calculate the corrected specification temperature (See Section 11.1).

9.3.5. After the test cup temperature has stabilized at the specification or target flash point, fill the syringe with the sample to be tested and insert the syringe into the filling orifice taking care not to lose any sample. Discharge the specimen into the test cup by depressing the syringe plunger to its lowest position, and then remove the syringe. If the material has a viscosity greater than 45 SUS at 100 °F (38 °C) or equivalent of 9.5 cSt at 77 °F (25.0 °C), discharge the contents of the syringe directly into the cup. Immediately close the lid and shutter assembly.

9.3.6. Set the timer by pressing the switch to "1 min". In the meantime, open the gas control valve and light the pilot and the test flames. Adjust the test flame size with the pinch valve so as to match the size of the 4 mm diameter flame gage, which is located on the cover of the lid and shutter assembly.

9.3.7. After 1 min has elapsed, observe the temperature. If it is at the specification temperature (accounting for the differences of the barometer reading from 760 mm Hg), apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2.5 s, and watch for a flash.

9.3.7.1. **Caution:** The slide mechanism should not be pulled back too far. This will cause the mechanism to come undone. See the primary analyst or group leader to repair the slide mechanism if this occurs.

9.3.8. The material is considered to have flashed only if a comparatively large blue flame appears and propagates itself over the surface of the liquid. Occasionally, particularly near the actual flash point temperature, application of the test flame may give rise to a halo; this should be ignored.

9.3.9. Turn off the test and pilot flames. Clean the apparatus in preparation for the next test.

9.4. Flash/No Flash Procedure - 32 °F (0 °C) to Ambient Temperature:

9.4.1. If the specification or target flash point is at or below ambient temperature, cool the sample to 10 °F to 20 °F (5 °C to 10 °C) below that point by some convenient means.

9.4.2. Cool the tester to approximately the temperature of the sample by inserting the cooling block filled with a cooling mixture into the sample well. Dry the cup with a paper tissue to remove any collected moisture prior to adding the sample.

9.4.3. **Caution:** Be careful in handling the cooling mixture and cooling block; wear gloves and goggles. Mixtures such as dry ice and acetone can produce severe frost bite.

9.4.4. Follow the steps in 9.3.

9.5. Finite Flash Point Procedure at Ambient Temperature to 230 °F (110 °C) - Preliminary or Trial Test (Range Finding)[5]:

9.5.1. Follow the steps in 9.3 omitting the barometric reading and using an estimated finite flash point instead of a specification flash point temperature.

9.5.2. After 1 min has elapsed, observe the temperature, apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of 2.5 s, and watch for a flash.

9.5.3. For samples analyzed under 40 CFR part 261.21, if no flash is observed at 140 °F (60 °C), the sample will be tested at 149 °F (65 °C). If no flash is observed, the result will be reported as no flash.

9.5.4. If a flash is observed in Section 9.5.2 proceed as follows:

9.5.4.1. Use a temperature of 9 °F (5 °C) lower than the temperature observed in Section 9.5.2. If a flash is still observed, repeat at 9 °F (5 °C) lower intervals until no flash is observed.

9.5.5. If no flash point is observed in Section 9.5.2 proceed as follows:

9.5.5.1. Using a test temperature of 9 °F (5 °C) higher than the temperature observed in Section 9.5.2. If no flash is observed repeat at 9 °F (5 °C) higher intervals until a flash is observed.

9.5.6. **Note:** Never make a repeat test on the same specimen. Always take a fresh portion for each test.

9.6. Finite Flash Point Procedure at Ambient Temperature to 230 °F (110 °C) - Finite Determination:

9.6.1. Stabilize the test cup temperature at the temperature at which no flash occurred previously (Section 9.5.4.1 or 9.5.5.1). Observe if a flash occurs at this temperature. If no flash occurs, increase the temperature at 1 °F (0.5 °C)

[5] See Appendix D, Section D.2 for an allowable deviation during range finding determinations.

intervals by making small incremental adjustment to the temperature control knob and allowing 1 minute intervals between each increment and the flash point test. Record the temperature at which the flash actually occurs. Record the barometric pressure. Turn off pilot and test flames and clean up tester.

9.7. Finite Flash Point Procedure at 32 °F (0 °C) to Ambient Temperature - Preliminary or Trial Test (Range Finding)[6]:

9.7.1. Cool the sample to 5 °F to 10 °F (3 °C to 5 °C) below the expected flash point.

9.7.2. Cool the tester to approximately the temperature of the sample by inserting the cooling block filled with a cooling medium into the sample well.

9.7.3. Insert the specimen as in 9.3. Set the 1-minute timing device. After 1 minute, apply the test flame by slowly and uniformly opening the slide fully and closing completely over a period of approximately 2.5 s, and observe for a flash. Record the temperature.

9.7.4. If a flash is observed, proceed as follows:

9.7.4.1. Take a new specimen and re-cool the sample cup to 9 °F (5 °C) below the temperature observed in 9.7.3. After 1 minute, check for a flash. If the material flashes repeat test at 9 °F (5 °C) lower intervals until no flash is observed.

9.7.5. If no flash point is observed, proceed as follows:

9.7.5.1. Using a test temperature of 9 °F (5 °C) higher than the temperature observed in 9.7.3. If no flash is observed, repeat at 9 °F (5 °C) higher intervals until flash is observed.

9.7.6. **Note:** Never make a repeat test on the same specimen. Always take a fresh portion for each test.

9.8. Finite Flash Point Procedure at 32 °F (0 °C) to Ambient Temperature – Finite Determination:

9.8.1. Repeat with a new specimen, cooling both sample and tester to the temperature at which the material did not flash (Section 9.7.4.1 or 9.7.5.1). After 1 minute observe if a flash occurs at this temperature, if not, increase the temperature at 1 °F (0.5 °C) intervals by making small incremental

[6] See Appendix D, Section D.2 for an allowable deviation during range finding determinations.

adjustments to the temperature control knob, allowing 1 minute between each increment and the test for the flash point. Record the temperature at which the flash actually occurs. Record the barometric pressure.

9.9. Clean Up of Apparatus and Preparation for Next Test:

9.9.1. To prepare for the next test, unlock the lid assembly of the tester and raise it to the hinge stop. Soak up liquid samples with an absorbent paper tissue and wipe dry. Clean the underside of the lid and filling orifice. A pipe cleaner may be of assistance in cleaning the orifice.

9.9.2. If the sample is a viscous liquid or contains dispersed solids, after soaking up most of the sample add a small amount of a suitable solvent for the sample to the cup and then soak up the solvent and wipe clean the interior surfaces of the cup with an absorbent tissue paper.

9.9.3. After the cup has been cleaned, its temperature may be rapidly increased to some stand-by value by turning the temperature control knob to an appropriate point. It is convenient to hold the test cup at some stand-by temperature (depending on planned usage) to conserve time in bringing the cup within the test temperature range. The cup temperature may be quickly lowered by inserting the aluminum cooling block filled with an appropriate cooling mixture into the cup.

9.9.4. The syringe is easily cleaned by filling it several times with acetone or any compatible solvent, discharging the solvent each time, and allowing the syringe to air dry with the plunger removed. Replace the plunger, and pump several times to replace any solvent vapor with air.

10. QUALITY CONTROL:

10.1. Users of this method must operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as continuing checks on performance. The user is required to maintain performance records that define the quality of the data that are generated. Limits used to accept results are set in the reference method. Calculate historical limits for information only.

10.2. For information on corrective actions refer to the Corrective Actions section in the Chicago Regional Laboratory (CRL) Quality Management Plan (QMP). In the subsections of this section immediate corrective actions are discussed for specific QC audits.

10.3. Maintain all determination and analysis information in LIMS and a data package file.

10.4. ASSESSING LABORATORY PERFORMANCE:

10.4.1. Instrument Performance Check (IPC) Solution:

10.4.1.1. Check the performance of the tester by determining the flash point of the p-xylene reference standard (Section 7.1) in duplicate on a daily basis when in use.

10.4.1.2. The mean of the results should be 81 °F ± 1.5 °F (27.2 °C ± 0.8 °C).

10.4.1.3. If the results are outside the limits, remove the thermometer and observe whether sufficient heat transfer paste surrounds the thermometer to provide good heat transfer from the cup to the thermometer. Ensure that the Setaflash tester and barometer are both functioning properly. Repeat the analyses of the IPC solution.

10.5. ASSESSING ANALYTE RECOVERY AND DATA QUALITY:

10.5.1. Laboratory Duplicate (DUP):

10.5.1.1. Samples selected for duplicate analysis are designated by the sampling organization or analyst if no designation is made.

10.5.1.2. If no designation is made by the sampling organization, a minimum of one duplicate must be analyzed for each sampling site.

10.5.1.3. Analyze at least one duplicate for each group of 10 samples or fewer collected. (Example: 11 collected samples require 2 duplicates).

10.5.1.4. The sample aliquot taken for duplicate analysis must be from the same bottle as the sample.

10.5.1.5. For this SOP a sample result (S) is considered to be the mean of two flash point determinations. A duplicate result (DUP) is considered to be the mean of two additional flash point determinations.

10.5.1.6. Analyze the sample (S) and duplicate (DUP).

10.5.1.7. Independently calculate the mean of the sample (S_{mean}) and the mean of the duplicate (DUP_{mean}):

$$S_{mean} \text{ or } DUP_{mean} = (FP_1 + FP_2)/2$$

Where
S_{mean} or DUP_{mean} = the average flash point
FP_1 = first flash point determination
FP_2 = second flash point determination

10.5.1.8. Evaluate the absolute difference (Δ) of the sample mean (S_{mean}) and duplicate mean (DUP_{mean}) as follows:

$$\Delta = \left| S_{mean} - DUP_{mean} \right|$$

Where
Δ = the absolute difference of the flash point
S_{mean} = mean sample flash point
DUP_{mean} = mean duplicate flash point

10.5.1.9. If the absolute difference (Δ) exceeds 3 °F (1.7 °C), consider the results suspect.

10.5.1.10. Take the following corrective action. Verify that the aliquots taken are from the same phase and that all the instruments are working properly. Make sure that there is no excessive draft in the test area, the sample and duplicate sample sizes are consistent, and that the sample cup is clean. Repeat the analysis.

10.5.1.11. If the mean of the result is within the limits, no further action is required.

10.5.1.12. If the mean of the determinations is outside the limits and results are to be reported, the Group Leader will be notified with the analyst's recommendation for a final evaluation of the data.

10.6. Quality Control Summary:

AUDIT	FREQUENCY	LIMIT	ACTION
SRM:	Daily, before	81 °F ± 1.5 °F	Reviewed April 2018

AUDIT	FREQUENCY	LIMIT	ACTION
p-xylene, the mean of two determinations	samples	(27.2 °C ± 0.8 °C)	
Sample: Absolute Difference (Δ) between two finite determinations	Every sample	Δ ≤ 2 °F (1 °C)	Reviewed April 2018
Duplicate: Absolute Difference (Δ) between the mean of a sample and the mean of a duplicate determination	One per sampling site, or at least every 10 samples	Δ ≤ 3 °F (1.7 °C)	Reviewed April 2018[7]

11. DATA & RECORDS MANAGEMENT:

11.1. Correction for Barometric Pressure:

11.1.1. When the barometric pressure differs from 760 mm Hg (101.3 kPa), calculate the flash point-temperature by means of the following equations:

Calculated Flash Point

$$°F = F + 0.06\,(760 - P)$$

$$°C = C + 0.03\,(760 - P)$$

Where:

F = Observed flash point in °F.
C = Observed flash point in °C
P = Barometric pressure (mm Hg).

11.1.2. Likewise determine the corrected specification flash point by the following equation:

$$F = S - 0.06\,(760 - P)$$

[7] ASTM D3278-78 Section 15.2.1 established the criteria for repeatability between the same operator on different days at ≤ 3 °F (1.7 °C). CRL has adopted these criteria for duplicate determinations.

$$C = S - 0.03\,(760 - P)$$

Where:

F = Flash point to be observed to obtain the corrected specification flash point in °F.
C = Flash point to be observed to obtain the corrected specification flash point in °C
S = Specification flash point (°F or °C).
P = Barometric pressure (mm Hg).

11.1.3. **Note:** the Traceable® Barometer (Section 6.1.3) reads in units of millibar (mb) or inches of mercury (in Hg). Use the following equations to convert the barometer reading into mm Hg for calculating equations 11.1.1 and 11.1.2.

11.1.3.1. $P_{mmHg} = 25.4 \times P_{inHg}$

11.1.3.2. $P_{mmHg} = 0.750062 \times P_{mb}$

11.1.4. See Appendix A, Section A.1.1.2 for an approved calculation spreadsheet.

11.2. When using the flash/no flash method, report whether the sample flashed at the required flash point and that the flash/no flash method was used.

11.3. If an actual flash point was determined, report the mean of duplicate runs to the nearest 1 °F (0.5 °C), provided the difference between the two values does not exceed 2 °F (1 °C).

11.3.1. Calculate the mean of the sample (S_{mean}) as follows:

$$S_{mean} = (FP_1 + FP_2)/2$$

Where
S_{mean} = the average flash point
FP_1 = first flash point determination
FP_2 = second flash point determination

11.4. Raw data and bench sheets are to be submitted with the data package

11.4.1. See Appendix A, Section A.1.1 for bench sheet information.

11.5. Any irregularities in labeling or preservation of samples, or other unusual observations, must be documented in a case narrative and brought to the attention of

the data user.

11.6. All electronic records associated with any data package generated must be archived following CRL.SOP AIG055.

11.7. All reviews are to be performed following this analytical procedure (CRL.SOP AIG048A) and data verification procedure (CRL.SOP AIG055).

11.8. Only final reports generated through LIMS are transmitted to the client. Raw data can be transmitted upon request.

12. TROUBLESHOOTING:

12.1. Refer to Section 5 Cautions and Interferences for common problems and resolutions.

12.2. Refer to the manufacturer's instrument manuals for equipment specific troubleshooting. The manuals are located with the instrument.

13. PREVENTATIVE MAINTENANCE:

13.1. Document all preventive maintenance in the instrument logbook describing the nature of the problems, steps taken to resolve the problems, and final solutions. The logbook is located with the instrument.

13.2. Refer to the manufacturer's instrument manuals for equipment specific preventive maintenance. The manuals are located with the instrument.

14. REFERENCES:

14.1. ASTM D 3278 - 78, Standard Test Methods for Flash Point of Liquids by Setaflash Closed Tester.

14.2. "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods", EPA Publication SW-846, Method 1020B, Revision 2, November 2004. Accessed on-line March 30, 2018: https://www.epa.gov/sites/production/files/2015-12/documents/1020b.pdf

14.3. Rapid Tester® Model RT-1, Operation and Service Technical Manual

14.4. Element LIMS™ Version 6 from Promium®, "User Manual", Promium, LLC 2013.

14.5. Element Data System™ Data Tool™ Version 3.590, "New User Tutorial", Promium, LLC 1999.

14.6. Pressure Conversion, Accessed on-line April 12, 2016: http://www.srh.noaa.gov/images/epz/wxcalc/pressureConversion.pdf

15. REVISION HISTORY:

V = Qualtrax Version, S = Status, R = Revision

Note: Description of changes only summarize significant changes. Minor changes not recorded in the revision history include, but are not limited to, addition of general information, correcting grammar, spelling, formatting, section changes, or re-wording for clarity that do not affect the meaning of a sentence.

V	S	Location & Description of Change History
5	R	This SOP was revised for the 2018 cycle according to GEN006 Version #4, QAC CRL Document # CHKLST 001A Version #6, and 2017 Policy Implementation Checklist (2017 PIL).
		Cover Page: Page 1: Added "based on" SW-846 Method 1020B and replaced LAB logo with ANAB logo.
		Section 6.1.1: Replaced "weighing bottle" with "tared small beaker or disposable solo cup".
		Section 10.6. Changed all action status to Reviewed April 2018.
		Section 11.6: Changed archiving procedure from CRL.SOP GEN001 to CRL.SOP AIG055.
		Sections 11.7 and A.2.3: Changed data review procedure from CRL.SOP GEN015 to CRL.SOP AIG055
		Draft Document workflow instance Qualtrax # 13801
4	R	Section 9.5, 9.7, Appendix D.2: Added footnotes to range finding instructions (9.5 & 9.7) to see Appendix D.2, which describes a technically justifiable deviation for range finding. This resolves CAR ID # 9215.
3	R	This SOP was revised for the 2016 cycle according to GEN006 Version #3, QAC CHKLST-001 V7.0 Modified 01-14-15, and SOP CHKLST-001A v2. Revisions in this SOP version also address corrective action report (CAR) ID# 4982.
		4.2: added SDS statement.
		6.1.1.1: added instructions to check the syringe daily when in use before analysis.
		6.1.3: Updated the equipment description for the barometer, and the certification frequency.
		6.1.4: added analytical balance to equipment list.
		6.1.5: added weight set to equipment list.
		9.1.1.1: added instruction to check syringe capacity.
		10.4.1.1: added clarification that the p-xylene check is performed on a daily basis when in use.

V	S	Location & Description of Change History
		11.1.3: Added conversion factor equations for converting pressure units from the barometer into mm Hg for flashpoint calculations.
		14: Reviewed/updated references, and added reference source for the pressure conversions used in this SOP.
		Appendix A.1.1.2: Updated the information for the flashpoint benchsheet and calculations file, which is now located in Qualtrax.
		Appendix D.2: Remove a deviation that is now covered in Sections 6.1.1.1 and 9.1.1.1. concerning syringe recertification (checking) requirement.
2	R	L-A-B logo was added.
1	R	Entire Document: Reorganized and renamed sections, including the addition/deletion of sections; reformatted to include subsection numbering system; revised with editorial changes to comply with CRLSOP GEN006 Revision 7.1. Edits to this version are too numerous to summarize here, see the hardcopy Track Changes Document filed with this revision. Section 1.9: Added the measurement uncertainty is set by the reference method (ASTM D3278-78). Section 6.1: Thermometer and barometer were added to the calibrated support equipment. Section 10.5.1: Clarified the requirements for duplicate analysis. Added calculations for determining sample and duplicate mean, and evaluating the sample and duplicate absolute difference. Section 10.5.1.9: Updated the sample and duplicate absolute difference result to that which is allowed in the reference method (ASTM D3278-78). Section 10.6: Added a Quality Control Summary chart. Section 11.3.1: Added the calculation for determining the mean of two finite flash point determinations. The mean is the value reported. Section 14: References were reviewed for applicability. Those cited are current to date. Section 15: Started a Revision History Appendix B: Added procedures from the reference method (ASTM D3278-78) for testing high viscosity liquids. Appendix C: Added procedures on how to address triplicate analyses for CIS samples. Appendix D: Deviations in this SOP from ASTM D3278-78 are addressed here.

APPENDIX A: LIMS Entry and Reporting

A. CRL uses Element DataSystem (ElmNT). Refer to References 14.4 and 14.5.

A.1. **Creating a Batch and Benchsheet:**

A.1.1. Create a batch and benchsheet describing the sample preparation procedure:

A.1.1.1. Use template "bch_C_A&IGeneric_v#.rpt".

A.1.1.2. Also use the following file to print a benchsheet to collect flashpoint data and to perform flashpoint calculations:

Qualtrax → Documents → US EPA R5 CRL → A&I → Spreadsheets → SS-AIG048A-Benchsheet_&_Calcs.xlsm.

A.1.2. Refer to Section 10 of this SOP for QC requirements, and Section 10.6 for a summary of audit types and frequency.

A.1.3. The preparation date in LIMS should match the actual preparation date on the laboratory benchsheet.

A.1.4. **Note:** By convention, if the sample preparation proceeds overnight, the date started is used for the LIMS preparation date.

A.1.5. Save the batch and benchsheet information in the LIMS database.

A.2. **Data Entry:**

A.2.1. From the ElmNT pull down menu, select **Laboratory**, then **Data Entry/Review**, and the **batch** created in section A.1.

A.2.2. Data is entered manually. Enter all pertinent data into the LIMS database.

A.2.3. When all data are entered, click the **Save** button on the top row. After saving, proceed to the Review page by clicking **Query** on the second row. Verify that all conversions to reporting units and dilutions have been calculated correctly. Verify that reporting limits have been correctly applied. Flags may be added at this stage, following the guidance given in SOP AIG055.

A.2.4. Before review by the peer, the data must be locked and updated to

Analyzed.

A.3. **Report Generation:**

A.3.1. Preparation of a Draft Report:

A.3.1.1. Ensure that all data are entered with the status of Analyzed.

A.3.1.2. From ElmNT pull down menu, select **Project Management** and then **Reports**.

A.3.1.3. Choose the work order number, analysis, and report format. Select Draft report.

A.3.1.4. **Note:** This draft report need not be signed. It is only for the purpose of review.

A.3.1.5. Submit the draft report with the data package to a peer reviewer.

A.3.1.6. After completing the data review, the peer reviewer updates the status of the LIMS entries to **Reviewed**.

A.3.2. Preparation of a Final Report:

A.3.2.1. After the peer reviewer has updated the status of the LIMS entries to **Reviewed**, a final report may be generated.

A.3.2.2. Ensure that all data are now in **Reviewed** status. Select **Final Report** or **Modified Final Report**. All pages of the report and the transmittal form must be signed and dated by the analyst.

APPENDIX B: Testing High Viscosity Liquids

B. ASTM D3278-78 A4: TESTING HIGH VISCOSITY LIQUIDS

B.1. High-viscosity materials may be added to the cup by the following procedure:

B.2. Back load a 5 or 10 mL syringe with the sample to be tested and extrude 4 mL into the cup. Spread the specimen as evenly as possible over the bottom of the cup.

B.3. If the sample cannot be loaded into a syringe and extruded, other means of adding the sample to the cup may be used such as a spoon. Add approximately 4 mL of material to the spoon and then push the material from the spoon into the cup.

B.4. If the test specimen does not close the sampling port in the cup, seal the cup externally by suitable means.

B.5. Using the appropriate procedure, determine the flash point of the specimen which has been added to the tester.

APPENDIX C: Testing Criminal Investigation Samples (CIS)

C. Testing CIS samples under CRL SOPGEN007:

C.1. The following criteria are followed in regards to flash point determinations for CIS samples requiring triplicate analysis.

C.2. For finite flash point determinations, triplicate flash points are determined by first performing a range finding determination. Then three finite flash point determinations are made.

C.3. Use the first and second determination to calculate the absolute difference of the flash point determinations, and apply the limit that the difference between the two values does not exceed 2 °F (1 °C).

C.4. Report each triplicate result independently to the nearest 1 °F (0.5 °C), provided the difference between the first and second determination does not exceed 2 °F (1 °C).

C.5. For flash/no flash determinations, triplicate flash points, are determined by performing three flash point determinations at the specified flash/no flash temperature.

APPENDIX D: Deviations from the Reference Method

D. Deviations in this SOP from ASTM D3278-78:

D.1. Section 1.2, 9.3.5, and Appendix B: CRL does not perform viscosity determinations. Free flowing liquids that are readily drawn into the syringe and discharged through the filling orifice are injected through the filling orifice. A sample that is drawn into the syringe but not readily discharged through the filling orifice may be injected directly into the cup, as allowed in ASTM D3278-78, Section 8.4. For testing high viscosity liquids Annex 4 of ASTM D3278-78 is followed (See Appendix B of this SOP).

D.2. Section 9.5 and 9.7: The reference method calls for increasing or decreasing the temperature in increments of 9 °F during range finding. This SOP allows the range finding temperature to change in increments of 9 ± 1 °F. This allows the Analyst to perform range finding more efficiently. For example, if the Analyst is increasing the temperature during range finding and the test cup temperature stabilizes at an increment of + 10 °F, then the Analyst may proceed with the determination instead of cooling down the apparatus and re-stabilizing the temperature. This deviation is technically justified because range finding does not impact the final result. Once a flash is observed the test is repeated at the last increment where no flash was observed, and then changed in increments of 1 °F.
Note: The thermometer used in this SOP has a Fahrenheit scale, therefore, only increments in °F are applicable.